Figure 2H

Supporting Information for Szyperski *et al.* (2002) *Proc. Natl. Acad. Sci. USA* **99** (12), 8009–8014. (10.1073/pnas.122224599).

Supporting Figure 9

Fig. 9. Experimental scheme for the 3D HCCH-COSY experiment. Rectangular 90° and 180° pulses are indicated by thin and thick vertical bars, respectively, and phases are indicated above the pulses. Where no rf phase is marked, the pulse is applied along *x*. The scaling factor k for ^{1}H chemical shift evolution during t_1 is set to 1.0. The high power 90° pulse lengths were: 5.8 ms for ^{1}H and 15.4 ms for ^{13}C, and 38 ms for ^{15}N. The lengths of the ^{1}H spin-lock purge pulses are: first SL_x, 2.8 ms; second SL_x, 1.7 ms; SL_y, 4.9 ms. SEDUCE is used for decoupling of ^{13}CO during t_1 and t_2 (rf field strength = 1 kHz). The WURST scheme is used for decoupling of ^{13}C during acquisition. The ^{1}H carrier is placed at the position of the solvent line at 0 ppm before the start of the first semiconstant-time ^{1}H evolution period, and then switched to the water line at 4.78 ppm after the second 90° ^{1}H pulse. The ^{13}C and ^{15}N rf carriers are set to 38 ppm and 120.9 ppm, respectively. The duration and strengths of the pulsed z-field gradients (PFGs) are: G1 (500 ms, 6 G/cm); G2 (500 ms, 7 G/cm); G3 (100 ms, 12 G/cm); G4 (100 ms, 12.5 G/cm); G5 (2 ms, 9 G/cm);G6 (500 ms, 5 G/cm); G7 (1.5 ms, 8 G/cm); G8 (400 ms, 6 G/cm). All gradients are applied along z axis and are of rectangular shape. All PFG pulses are of rectangular shape. A recovery delay of at least 100-ms duration is inserted between a PFG pulse and an rf pulse. The delays are: t_1 = 1.6 ms, t_2 = 850 ms, t_3 = 2.65 ms, t_4 = 3.5 ms, t_5 = 7 ms, t_6 = 1.6 ms, t_7 = 3.2 ms. Phase cycling: $f_1 = x$; $f_2 = x, -x$; $f_3 = x, -x$; $f_4 = x$; f_5(receiver) = $x, -x$. Quadrature detection in $t_1(^{13}C/^{1}H)$ and $t_2(^{13}C)$ is accomplished by altering the phases f_2 and f_3, respectively, according to States-TPPI. Water suppression is accomplished by coherence pathway rejection using spin-lock purge pulses and pulsed field z-gradients. For acquisition of central peaks derived from ^{13}C steady state magnetization, a second data set with $f_1 = -x$ is collected. The sum and the difference of the two resulting data sets generate subspectra II and I, respectively, containing the central peaks and peak pairs.

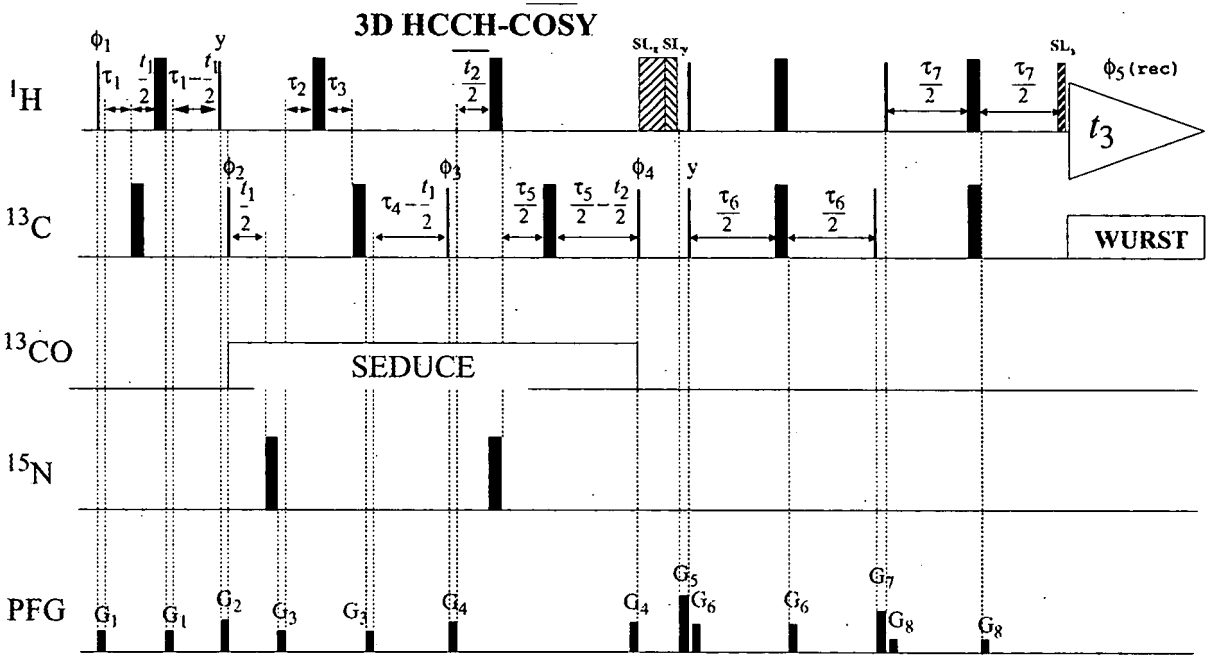
3D HCCH-COSY
^{1}H
^{13}C
^{13}CO
^{15}N
PFG
WURST
SEDUCE
t_3
ϕ_1
ϕ_2
ϕ_3
ϕ_4
ϕ_5 (rec)
y
τ_1
$\frac{t_1}{2}$
$\tau_1-\frac{t_1}{2}$
τ_2
τ_3
$\frac{t_2}{2}$
$\tau_4-\frac{t_1}{2}$
$\frac{\tau_5}{2}$
$\frac{\tau_5}{2}-\frac{t_2}{2}$
$\frac{\tau_6}{2}$
$\frac{\tau_7}{2}$
SL$_x$ SL$_y$
SL$_z$
G$_1$
G$_2$
G$_3$
G$_4$
G$_5$
G$_6$
G$_7$
G$_8$

Figure 9